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FINAL REPORT

U. S. ATOMIC ENERGY COMMISSION CONTRACT AT- (40-1)-2670

Project Title:

Radiochemistry as Applied to Geochemical

Problems: Neutron Activation Analysis

Project Director:

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Duration of Contract:

January 1, 1960 - December 31, 1971.

Transmitted by:

James Y. McDonald, Executive Director

The University of Kentucky Research Foundation

Date Transmitted:

1-14-72

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I. INTRODUCTION

This report is the final report of the project "Radio-chemistry as Applied to Geochemical Problems: Neutron Activation Analysis." This project has been supported by the U. S. Atomic Energy Commission during the period Jan. 1, 1960 through Dec. 31, 1971 under Contract AT-(40-1)-2670.

The report is divided into two basic sections:

- A. A review of the accomplishments of the project over
 the entire active period. Included are lists of
 publications supported by this project, ORO Technical
 Reports submitted, and advanced degrees granted which
 resulted from research supported, at least in part,
 by this contract.
- B. A more specific report on the work accomplished during the final contract year, 1971. Included are brief descriptions of the research work completed during the year, continuing research, academic progress, a financial statement for the year, and related contractual statements.

Copies of all publications and manuscripts submitted for publication have been previously submitted to Dr. Dent C. Davis of the Oak Ridge Operations Office and the Division of Research of the U. S. Atomic Energy Commission in Washington, D. C. Therefore, copies of these materials are not included with this report, but lists are provided in Sections II and III of this report. Specific technical reports are included only

for research conducted in the last year (1971) of this project.

These specific reports are given in Section V-A of this document.

A brief summary of the accomplishments of the project More than 50 publications in the fields of activation analysis and the geochemistry of the meteorites have resulted directly from the research supported by this contract. group is one of the leaders in the determination of meteoritic elemental abundances and many of the generally accepted "cosmic abundances" for the elements are based on our studies. addition, the group has been among the leaders in the development and application of 14 MeV neutron activation analysis and coincidence counting techniques as applied to activation analysis. The foundation of work developed under U.S.A.E.C. support led to the selection of this group by N.A.S.A. to participate in the analyses of the lunar rocks returned by the The contract director has presented invited Apollo program. papers at international meetings in Canada, England, Germany, Norway and Australia based on contract work. Most recently, the author presented two invited papers and chaired the opening session of the N.A.T.O. Symposium, Activation Analysis in Geochemistry and Cosmochemistry in Oslo, Norway.

The project has supported numerous graduate students in their theses or dissertation research, resulting in the granting of 3 M. S. degrees and 10 Ph.D. degrees. In addition, one M. S. candidate and two Ph.D. candidates are currently still at work on projects originally supported by this contract.

Numerous undergraduate students have worked as technicians on this project and have gone on to advanced degrees at institutions such as Carnegie Mellon University, Oregon State University, the University of Wisconsin, and others.

It is felt that this project has been productive over the years and has contributed significantly in the development of new approaches to activation analysis and to an understanding of the chemistry of the meteorites. It is somewhat ironic to note that the terminal years (1970,1971) of the project were the most productive years in terms of papers published, degrees granted and oral papers presented. These two years saw 21 papers published or in press, more than 20 oral papers presented, and three Ph.D. degrees completed while the contract budgets were among the lowest in the eleven years of the contract. While it was hoped that U.S.A.E.C. support could have been continued based on this record of accomplishment, the author does wish to express his gratitude to the U.S.A.E.C. for their support and encouragement over the past decade.

II. PUBLICATIONS SUPPORTED BY CONTRACT AT-(40-1)-2670 1960-1971

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- 1. W. D. Ehmann, Recent Improvement in our Knowledge of Cosmic Abundances, J. Chem. Ed. 38, 53-57, (1961).
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- 6. A. Amiruddin and W. D. Ehmann, Tungsten Abundances in Meteoritic and Terrestrial Materials, Geochim. Cosmochim. Acta 26, 1011-1022, (1962).

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9. J. L. Setser and W. D. Ehmann, Zirconium and Hafnium Abundances in Meteorites, Tektites and Terrestrial Materials, Geochim. Cosmochim. Acta 28, 769-782, (1964).

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- 11. P. A. Baedecker and W. D. Ehmann, The Distribution of Some Noble Metals in Meteorites and Natural Materials, Geochim. Cosmochim. Acta 29, 329-342, (1965).
- 12. J. R. Vogt and W. D. Ehmann, Silicon Abundances in Stony Meteorites by Fast Neutron Activation Analysis, Geochim. Cosmochim. Acta 29, 373-383, (1965).
- 13. J. R. Vogt, W. D. Ehmann and M. T. McEllistrem, An Automated System for Rapid and Precise Fast Neutron Activation Analysis, Int. J. Appl. Radiat. Isotopes 16, 573-580, (1965).
- 14. J. R. Vogt and W. D. Ehmann, An Automated Procedure for the Determination of Oxygen Using Fast Neutron Activation Analysis; Oxygen in Stony Meteorites, Radiochim. Acta 4, 24-28, (1965).

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- 15. J. R. Vogt and W. D. Ehmann, The Non-Destructive Determination of Silicon and Oxygen in Meteorites by Fast Neutron Activation Analysis, (Paper presented at the 1965 International Conference on Modern Trends in Activation Analysis), Texas A. and M. Press, pp. 82-85, (1966).
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- 21. K. W. Lieberman and W. D. Ehmann, Determination of Bromine in Stony Meteorites by Neutron Activation, J. Geophys. Res. 72, 6279-6287, (1967).

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- 36. W. D. Ehmann and J. W. Morgan, Precise Non-Destructive Determination of Some Major Elements in Lunar Materials by 14 MeV Neutron Activation, Proceedings of the 2nd Oak Ridge Conference on the Use of Small Accelerators for Teaching and Research, March 23-25, 1970, U.S.A.E.C. Document CONF-700322, pp. 205-220, (1970).
- 37. W. D. Ehmann, D. E. Gillum, J. W. Morgan, R. A. Nadkarni, T. V. Rebagay, P. M. Santoliquido, and D. L. Showalter, Chemical Analyses of the Murchison and Lost City Meteorites, Meteoritics 5, 131-136, (1970).

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- 39. W. D. Ehmann and D. L. Showalter, Elemental Abundance Trends in the Australite Strewn Field by Non-Destructive Neutron Activation, Proceedings of the N.A.T.O. Advanced Study Institute-Activation Analysis in Geochemistry and Cosmochemistry, Kjeller, Norway, September 7-12, 1970, Universitetsforlaget, Os.o, pp. 253-260, (1971).
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- 41. J. W. Morgan and W. D. Ehmann, 14 MeV Neutron Activation Analysis of Rocks and Meteorites, Proceedings of the N.A.T.O. Advanced Study Institute-Activation Analysis in Geochemistry and Cosmochemistry, Kjeller, Norway, September 7-12, 1970, Universitetsforlaget, Oslo, pp. 81-97, (1971).
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- 44. W. D. Ehmann, Antimony (51), Elemental Abundances in Meteorites, Ed. Brian Mason, International Association of Geochemistry and Cosmochemistry, In press.
- 45. W. D. Ehmann, Tantalum (73), Elemental Abundances in Meteorites, Ed. Brian Mason, International Association of Geochemistry and Cosmochemistry, In press.
- 46. W. D. Ehmann, Tungsten (74), Elemental Abundances in Meteorites, Ed. Brian Mason, International Association of Geochemistry and Cosmochemistry, In press.
- 47. W. D. Ehmann, Platinum (78), Elemental Abundances in Meteorites, Ed. Brian Mason, International Association of Geochemistry and Cosmochemistry, In press.
- 48. W. D. Ehmann, Gold (79), Elemental Abundances in Meteorites, Ed. Brian Mason, International Association of Geochemistry and Cosmochemistry, In press.
- W. D. Ehmann and D. E. Gillum, Platinum and Gold in Chondritic Meteorites, Chemical Geology, Accepted 1971.
- D. E. Gillum and W. D. Ehmann, Noble Metal Determination in Meteorites, Radiochim. Acta, Accepted (1971).
- P. M. Santoliquido and W. D. Ehmann, Bismuth in Stony Meteorites and Standard Rocks, Goochim. Cosmochim. Beta. Submitted Sept. (1971).
- (A list of some 30 additional publications not supported by this contract will be provided on request.)

ORO-2670-21	Abundances of Some Volatile Elements in Meteorites, 1967.
ORO-2670-22	Silicon Abundances in Some Meteorites and Standard Rocks by Activation Analysis, 1967.
ORO-2670-23	The Geochemistry of Br in Igneous Rocks and Deep-Sea Sediments, 1967.
ORO-2670-24	Progress ReportRadiochemistry as Applied to Geochemical Problems; Neutron Activation Analysis, 1967.
ORO-2670-25	The Distribution of Some Noble Metals in Meteorites and Natural Materials, II, 1968.
ORO-2670-26	The Walltown, Kentucky Meteorite, 1968.
ORO-2670-27	Heat-sealed Polyethylene Sample Con- tainers for Neutron Activation Analysis, 1968.
ORO-2670-28	The Non-Destructive Determination of Iridium in Meteorites Using Gamma-gamma Coincidence Spectrometry, 1968.
OPO-2670-29	Progress ReportRadiochemistry as Applied to Geochemical Problems; Neutron Activation Analysis, 1968.
ORO-2670-30	Instrumental Activation Analysis of Meteorites Using Gamma-Gamma Coin- cidence Spectrometry, 1968.
ORO-2670-31	The Determination of Zirconium and Hafnium in Meteorites and Terrestrial Materials, 1969.
ORO-2670-32	Some Major and Trace Element Abun- dances in the Allende Meteorite by Activation Analysis, 1969.

ORO-2670-33	Progress ReportRadiochemistry as Applied to Geochemical Problems; Neutron Activation Analysis, 1969.
ORO-2670-34	Simultaneous Determination of Zir- conium and Hafnium in Standard Rocks by Activation Analysis, 1969.
ORO-2670-35	Multiparameter Coincidence Spectro- metry Applied to the Non-Destructive Neutron Activation Analysis of Meteorites, 1969.
ORO-2670-36	Zirconium and Hafnium in Meteorites by Activation Analysis, 1969.
ORO-2670-37	Precise Non-Destructive Determination of Some Major Elements in Lunar Material by 14 MeV Activation Analysis, 1970.
ORO-2670-38	Instrumental Activation Techniques for the Analysis of Meteorites and Lunar Materials, 1970.
ORO-2670-39	Chemical Analyses of the Murchison and Lost City Meteorites, 1970.
ORO-2670-40	Progress Report Radiochemistry as Applied to Geochemical Problems; Neutron Activation Analysis, 1970.
ORO-2670-41	Coincidence Counting Applied to the Activation Analysis of Meteorites and Rocks, 1970.
ORO-2670-42	Elemental Abundance Trends in the Australite Strewn Field by Non-Destructive Neutron Activation, 1970.
ORO-2670-43	14 MeV Neutron Activation Analysis of Rocks and Meteorites, 1970.

ORO-2670-44	Platinum and Gold in Chondritic
ORO-2670-45	Meteorites, 1971. Noble Metal Determination in Meteorites, 1971.
ORO-2670-46	Bismuth in Stony Meteorites and Stan- dard Rocks, 1971.
ORO-2670-47	This report, 1971.

IV. THESES AND DISSERTATIONS SUPPORTED IN PART BY THIS CONTRACT

M. S. Theses

- Peter Roys Rushbrook (M. S. 1960)
 "Iridium in Stone Meteorites by Neutron Activation Analysis."
- James L. Setser (M. S. 1963)
 "Determination of Zirconium and Hafnium in Meteorites, Sediments, and Terrestrial Materials by Neutron Activation Analysis."
- 3. Philip A. Baedecker (M. S. 1964)
 "Platinum, Gold, and Mercury in Meteorites and Natural
 Materials by Neutron Activation Analysis."

Ph. D. Dissertations

- Achmad Amiruddin (Ph.D. July, 1961)
 "Tungsten Abundances in Terrestrial and Extra-Terrestrial Materials by Neutron Activation Analysis."
- James Thomas Tanner (Ph.D. Sept., 1966)
 "The Determination of Antimony in Natural Materials by Neutron Activation."
- Kenneth W. Lieberman (Ph.D. July, 1966)
 "The Determination of Bromine in Terrestrial and Extraterrestrial Materials by Neutron Activation Analysis."
- 4. James R. Vogt (Ph.D. April, 1966) "The Non-Destructive Determination of Silicon and Oxygen in Meteorites by Fast Neutron Activation Analysis."
- 5. Philip Ackerman Baedecker (Ph.D. 1967)
 "The Distribution of Gold and Iridium in Meteoritic and Terrestrial Materials."
- 6. Teofila V. Rebagay (Ph.D. 1969)
 "The Determination of Zirconium and Hafinium in Meteorites and Terrestrial Materials by Activation Analysis."
- 7. David M. McKown (Ph.D. 1969)

 "Application of Gamma-Gamma Coincidence Counting Techniques to the Non-Destructive Activation Analysis of Meteoritic Materials."
- Donald L. Showalter (Ph.D. 1970)
 "Compositional Trends in Australites, Impact Glasses, and Associated Natural Materials by Activation Analysis."

Ph. D. Dissertations, cont'd

- 9. David E. Gillum (Ph.D. 1971)
 "Group Separation Techniques for the Determination of Noble Metals in Meteorites by Activation Analysis."
- 10. Patricia M. Santoliquido (Ph.D. 1971)

 "The Determination of Bismuth in Meteorites and Rocks by Neutron Activation Analysis."

Graduate Work in Progress Supported by this Contract

- 1. Michael D. Miller
 "Coincidence Techniques in Activation Analysis and
 Computer Resolution of Activation Data," Ph.D. program.
- 2. Chung Lu Sya
 "The Use of 252Cf in Activation Analysis and Capture
 Gamma-Ray Activation Analysis," Ph.D. program.
- 3. Patricia A. Kumar
 "Ground Water Geochemistry Employing Activation Analysis,"
 M.S. program.

V. SPECIFIC REPORT FOR THE PERIOD (January 1, 1971 - December 31, 1971)

Preprints and reprints of research conducted during this period have been submitted previously to the U.S.A.E.C. (ORO Reports 2670-41,42,43,44,45 and 46 and reprints bearing dates 1970 and 1971 in Section II of this report). The material in these preprints and reprints will not be repeated in detail here. Only the research which has been just recently submitted for publication or is still in progress will be reviewed below.

Publications supported by this contract during the period since the last progress report (7 published, 8 in press, 2 submitted) are listed in Section II of this report. In addition the following list of oral papers or talks were given by project personnel since submittal of our previous progress report ORO-2670-40, September, 1970. Those talks directly related to work supported by this contract are indicated with an asterisk.

- * 1. W. D. Ehmann, D. M. McKown, and J. W. Morgan, Coincidence Counting Applied to the Activation Analysis of Meteorites and Rocks, N.A.T.O. Advanced Study Institute -Activation Analysis in Geochemistry and Cosmochemistry, Kjeller, Norway, September 7-12, 1970. Invited paper.
- * 2. J. W. Morgan and W. D. Ehmann, 14 MeV Neutron Activation Analysis of Rocks and Meteorites, N.A.T.O. Advanced Study Institute Activation Analysis in Geochemistry and Cosmo-chemistry, Kjeller, Norway, September 7-12, 1970. Invited paper.
- * 3. W. D. Ehmann and D. L. Showalter, Elemental Abundance Trends in the Australite Strewn Field by Non-Destructive Neutron Activation, N.A.T.O. Advanced Study Institute Activation Analysis in Geochemistry and Cosmochemistry, Kjeller, Norway, September 7-12, 1970. Contributed paper.

- 4. W. D. Ehmann, Lunar Rocks, Eastern Kentucky University, October 22, 1970.
- * 5. W. D. Ehmann and D. E. Gillum, Determination of Noble Elements in Small Samples of Geochemical Interest, S.E./S.W. Am. Chem. Soc. Regional Meeting, New Orleans, La., December 3, 1970. Contributed paper.
- * 6. W. D. Ehmann, Meteorites and the Moon, Glendover School, Lexington, Ky., January 8, 1971.
 - 7. W. D. Ehmann and J. W. Morgan, Major Element Abundances in Apollo 12 Rocks and Fines by 14 MeV Neutron Activation, N.A.S.A. Apollo 12 Lunar Science Conference, Houston, Texas, January 11, 1971. Invited paper.
- * 8. W. D. Ehmann, Activation Analysis, Agronomy Seminar, University of Kentucky, January 29, 1971.
 - 9. W. D. Ehmann, Recent Developments in the Moon Program, Society of Sigma Xi, Lexington, Ky., February 25, 1971.
- *10. W. D. Ehmann, Meteorites and the Moon, Society for Applied Spectroscopy, Cincinnati Section, March 16, 1971.
- 11. W. D. Ehmann, Lunar Research, Glasgow Rotary Club, Glasgow, Ky., April 1, 1971.
- *12. W. D. Ehmann, Meteorites and the Moon, Western Kentucky University, Chemistry Department Seminar, April 2, 1971.
- *13. W. D. Ehmann, Meteorites and the Moon, Invited lecture to 1700 undergraduate students in Introduction Biology, University of Kentucky, October 20, 1971.

Specific technical reports on recently completed or continuing research follow:

A. Technical Reports

1. GROUP SEPARATION TECHNIQUES FOR THE DETERMINATION

OF NOBLE METALS IN METEORITES BY ACTIVATION ANALYSIS

David E. Gillum

A separation scheme has been developed for the determination of a series of noble metals in the same small geochemical sample. It was possible to determine iridium, cobalt, nickel, gold, platinum, silver, and ruthenium in a 100 mg meteorite sample. Iridium, cobalt, and nickel may be determined by non-destructive gamma-gamma coincidence counting, which avoids the problems associated with the radiochemical separation of iridium. Platinum was determined by counting 199 Au, which is the radioactive product from the β^- decay of 30 min. 199 Pt formed by the (n,γ) reaction on 198 Pt. This permits the determination of both gold and platinum while requiring the radiochemical separation of only one element A radiochemical separation for gold utilizing the technique of substoichiometric separation was developed. Gold was extracted with a 5×10^{-3} M solution of copper diethyldithiocarbamate in chloroform from a 1.5 M hydrochloric acid solution by shaking for 45 sec. This one step extraction for the radiochemical separation of gold has greater selectivity than most conventional separation techniques. Ruthenium was separated as the tetroxide by distillation from a sulfuric acid solution using sodium bromate as the oxidizing agent. After distillation, ruthenium was reduced to the metal with zinc. The radiochemical separation of silver was carried out by a rapid ion exchange procedure with Dowex 1-X8 resin.

Replicate analyses for gold and platinum were made on 39 selected chondritic meteorite samples. Replicate analyses for silver and ruthenium were made on 11 and 8 selected meteorites, respectively.

Gold and platinum were found to be positively correlated in all meteorites analyzed. These elements were also correlated with the metal phase content of the ordinary chondrites.

The atomic abundances of gold and platinum (relative to Si = 10⁶ atoms) for the Cl-C2-C3 carbonaceous chondrites were 0.19-0.20-0.18 and 1.34-1.07-1.43, respectively. Gold and platinum were found to be depleted in the L and LL group chondrites with respect to the carbonaceous chondrites, having an atomic abundance of 0.13 and 0.87 for the L group and 0.11 and 0.60 for the LL group, respectively. Atomic abundances for the H group chondrites were similar to those of the carbonaceous chondrites.

The enstatite chondrites were found to be depleted in the more refractory noble metals (iridium, osmium, and ruthenium) and enriched in the more volatile noble metals (gold and palladium) in comparison to the Cl carbonaceous chondrites. It is suggested that this elemental abundance pattern was established at the time of condensation of the enstatite chondrite parent bodies. A two stage condensation pattern is proposed. In the first stage, the more refractory noble metals would condense from the cooling solar nebula. Some process would then be required to remove this condensed fraction and the enstatite chondrite parent bodies would then condense from the residual material.

TABLE I
PLATINUM AND GOLD ATOMIC ABUNDANCES*

Class	Pt	Atomic	abundances (Si Au	= 10 ⁶ atoms)
Cl	1.37	[1.2]	0.19 [0.	19] 0.68
C2	1.07	[1.4]	0.20 [0.	19] 0.66
C3	1.43	[1.6]	0.18 [0.	17] 0.60
C4	1.60	-	0.13 [0.	13] -
All C	1.42	***	0.19 [0.	18] 0.64
All LL	0.60	[0.6]	0.11 [0.	12] 0.28
A11 L	0.87	[0.6]	0.13 [0.	13] 0.36
All H	1.26	[1.3]	0.18 [0.	19] 0.68
All E	1.34	[1.2]	0.29 [0.	26] 0.51

^{*}Literature values as compiled by Ehmann (1971 a,b) are given in brackets. L- and LL-groups were reported together as 0.6 for Pt.

References:

Ehmann W. D., et al. (1970), Geochim. Cosmochim. Acta 34,493-507.

Ehmann W. D., (1971 a,b), Papers in press.

Gillum D. E., (1971) Ph.D. Dissertation, University of Kentucky.

Note: More detailed accounts of this work appear in Reports ORO-2670-44,45 which were previously submitted to the U.S.A.E.C.

^{*}Non-destructive Ir data of Ehmann et al. (1970) on many of the same powders used in this work.

2. THE DETERMINATION OF BISMUTH IN METEORITES AND ROCKS

BY NEUTRON ACTIVATION ANALYSIS

Patricia M. Santoliquido

The abundance of bismuth is of great interest to cosmochemists for two reasons: bismuth is a pivotal point in theories of nucleosynthesis, since it is the last of the stable, naturally occurring elements and the end-product of the s-process; and because of its extreme volatility, it would be among the last of the elements to condense from the primordial broth, thus knowledge of its abundance could help elucidate the thermal history of the meteoritic parent bodies. However, there have been few previous determinations of bismuth in natural materials, because of the many difficulties inherent in determining an element present only at the parts per billion level in a highly complex matrix.

In the present work, bismuth has been determined by alpha counting of the Po-210 daughter activity of the Bi-210 formed by thermal neutron activation. Results are presented for thirty chondrites, six achondrites, eight separated meteoritic phases, and six U.S. Geological Survey standard rocks. The majority of the meteorite analyses were done in replicate. The rock analyses were done in triplicate. This represents the first analyses for bismuth of meteorites in the chondrite classes: C4, H4, E5, E6, and LL6 and considerably expands the achondrite work.

A number of trends which have been previously either noted or suspected by others have been further confirmed. The carbonaceous chondrites and ordinary chondrites show a decrease in bismuth content with increasing petrographic grade. The chalcophilic behavior of bismuth has been confirmed by correlation with sulfur content in the enstatite and ordinary chondrites.

The enstatite results show that the bismuth abundance is high for the trace element rich group and low for the trace element poor group, with a sharp hiatus between the two. These results also suggest that oldhamite may be an accessory host mineral for bismuth.

In general, the achondrites exhibit the same bismuth abundance level as the most metamorphosed ordinary chondrites. An exception is Kapoeta, in which bismuth is remarkably enriched.

The Allende phases show depletion of bismuth in the chondrules and white phase relative to the whole meteorite analyses. Allende black matrix was not analyzed separately, but it is evident from the other values that bismuth must be enriched there. This would lead to another case of enrichment in dark over light phase similar to those already noted for Pantar and Fayetteville.

Other separated phase work indicates that troilite is probably the principal host mineral for bismuth.

The bismuth abundances for the three ordinary groups, H, L, and LL, overlap each other to an exist suggests that accretion temperature rather than suggests that accretion temperatures have been condensation is the dominant factor affection between condensation temperatures have been condensation temperatures have been condensation that the H and L series from the abundance data. The turns indicate that: ordinary chondrites of the series formed in a slowly cooling zone of the series formation; ordinary chondrites of type I were formed in a slowly cooling zone of the meteorite parent condensation ordinary chondrites of type I were formed in a slowly cooling zone, where the two condensations were competitive.

lead to the estimate that enstatite bisneth and the system that was 35-55% matrix.

TABLE II

AVERAGE BISHUTH ABUNDANCES ST CLASSES TO

	Average for this work	Average (et all values	
Class	ppb		ACMINISTRA TOPARAMENT AND A STATE OF THE STA
Н3		31,3	3-6 (1)
H4	24.3	24.3	
H5	7.2		.€ - ♠ -
Н6			• •
L3		3 7 3	• •
L4	10.0		⊕ 1 •3
L5	7.0		3€ - 43
1.6			•

Basis of selection serial and an anna

This class her designed in

References:

Santoliquido P. M., (1971), Ph.D. Dissertation, University of Kentucky.

Note: A more detailed account of this work is given in Report ORO-2670-46, previously submitted to the U.S.A.E.C.

3. INTEGRATION OF THREE DIMENSIONAL DIGITAL PEAKS

IN MULTIPARAMETER COINCIDENCE SPECTROMETRY

Michael D. Miller

Coincidence spectrometry has been applied in the determination of elemental abundances by activation analysis. The chief advantage of coincidence spectrometry is its ability to reduce interferring activities from other isotopes when measuring the photopeak activity of an element exhibiting the simultaneous emission of two or more gamma rays, or a gamma ray in coincidence with annihilation radiation due to position emission.

In multiparameter coincidence spectrometry, a radioactive sample is placed between two detectors which are connected to the associated electronics as shown in Fig. 1.

The pulses from the detectors are amplified and passed through a single channel analyzer (eliminating the low energy noise) to a fast coincidence unit. Whenever pulses from the two detectors arrive simultaneously (within 15 ns.) at the fast coincidence unit, a gating pulse is sent to the multichannel analyzer which examines the energy of the pulses arriving from each detector.

The data are stored in a 64 x 64 matrix as shown in Fig. 2. The two independent variables plotted along the X-and Y-axes are the energies of the pulses in the two detectors at the time the gate was opened. The height of the Z-axis is the number of times the gate was opened when pulses with these energies were present.

The peaks located at (X,Y) equal to (1.17,1.33) and (1.33,1.17) are due to the simultaneous detection of one of the 60Co gamma rays in the first detector and the other gamma ray in the second detector. Since the volume of these peaks is proportional to the amount of 60Co present, it is important to be able to accurately determine either these volumes, or the ratio of these volumes to those of peaks produced by known amounts of 60Co counted under the same conditions.

The most common method for volume determination in the past has been spectrum stripping. In this method, known fractions of the peaks in the standard spectrum are repeatedly subtracted from the corresponding peaks in the sample spectrum. In addition to being both tedious and time consuming, the

accuracy of this method is limited by the skill of the operator in judging when the peak has been exactly subtracted out. In order to improve both the speed and accuracy of the volume determinations, it was desired that a numerical method of determining these volumes be developed.

Several numerical methods of two-dimensional peak integration have been investigated (1-4). The methods by Covell and Sterlinski appeared capable of being generalized for application to three dimensional peaks. With Covell's method, the integral of a two dimensional peak is given by

$$A_{n}^{C} = \begin{cases} k=+(n-1) \\ \sum_{k=-(n-1)}^{n} a_{k} \end{cases} - (n-1/2) (a_{n}+a_{-n}),$$

where n is the number of channels summed within the peak to the left or to the right of the center channel, a_o , and a_k is the number of counts in the kth channel.

Sterlinski's integral is given by

$$A_n^s = na_0 + \sum_{k=1}^n (n-2k+1/2)(a_k+a_{-k}).$$

This method is similar to Covell's except that increased weight is given to those channels near the center of the peak.

It can be seen that in Covell's method, the channels are summed out to the arbitrary channel n-1, and then the average number of counts in channels n and -n are subtracted from each channel in the original sum to allow for the background under the peak. This same approach may be used for three dimensional peaks. The channel closest to the peak center is first located. The distance of each channel from the center channel is then calculated, and those channels which arat the same distance from the center channel are summed to improve counting statistics. Dividing the sum of the counts at any given distance from the center channel by the number of channels contributing to that sum gives the average number of counts per channel at that distance. A graph of this quantity (Fig 3) mesembles half of a two-dimensional peak. As the distance from the center increases, the average number of counts per channel decreases until contributions from either the Compton scatter or from an interferring peak start being added to the sum. When such contributions do start being added to the sum, the average number of counts per channel increases. mum height of the curve represents the background under the peak, including possible contributions from the Compton scatter or interferring peaks. For the graphed data, the total number of counts in channels closer to the center than 5.05 channels is 2934 counts contained in 81 channels. Judging by the graph, the minimum on the curve is 7 counts with an expected error of ± 1.5 counts. Thus, the estimated background is $81 \times 7 = 567$ counts giving a resultant peak volume of 2934 - 567 = 2367

counts. If the error is 1.5 counts, the error in the background and consequently in the peak volume is $1.5 \times 81 = 122$ counts or about 5% of the peak volume. The statistically expected error, is about 1%; thus, this method of integration is unsatisfactory.

However, it is possible to use methods similar to Covell's and Sterlinski's to integrate the peak. If $C_{\rm i}$ is the sum of all the counts at a given distance from the center of the peak, $M_{\rm i}$ is the number of channels at that distance and i is an index such that $C_{\rm O}$ is the center channel, $C_{\rm l}$ is the sum of all the counts at the nearest distance, $C_{\rm l}$ is the sum of all the counts at the next nearest distance, etc., then the volume may be represented by

$$V_{n}^{c} = \sum_{k=0}^{n-1} C_{k} - \left\{ \sum_{k=0}^{n-1} M_{k} \times C_{n} \atop M_{n} \right\}$$

which is similar to Covell's formula, or by

$$V_{n}^{s} = \sum_{j=1}^{n} V_{j}^{c} = nc_{o} + \sum_{k=1}^{n} \left\{ n - k - \left\{ \sum_{j=0}^{k-1} M_{j} \right\} \times C_{k} \right\}$$

which is similar to Sterlinski's formula, or by

$$V_{n}^{m} = \sum_{k=0}^{n-1} \left\{ (n - k) C_{k} \right\} - \sum_{k=0}^{n-1} M_{k} \times C_{\underline{n}}$$

which is a modified version of Sterlinski's formula and is similar to a method by Wasson (4).

Each of these methods has been written into a computer program. The program estimates the peak center channel by least squares fitting the peak to an elliptic paraboloid and taking as the center channel the channel nearest the maximum of the fitted paraboloid. The program computes the integrals as a function of n by each of the three methods.

function of n by each of the three methods.

To test each of these methods a sample of 60 Co was counted eleven times using two 3" x 3" NaI(Tl) detectors. The spectra were analyzed by the computer program and the results of each of the methods for one of the coincident peaks is shown in Table 1. The results for the other peak are similar.

For both the 3-dimensional Covell and the 3-dimensional modified methods, the relative standard deviation decreased with increasing n to around n = 15 (corresponding to a distance of about 5 channels from the center, which is just off

the peak edge) and then increases with n. For the 3-dimensional Sterlinski method, the increase does not appear until much further out. The peak contains about 2300 counts, indicating that the error due to counting statistics alone is about 48 counts or approximately 2.1%. The best precision which one can reasonably expect to attain by any of the above methods is about 2.7% + .2%

In an attempt to improve the precision of these methods, another method was tried. It was observed that if the two peaks in a multiparameter spectrum were stacked one on top of the other by summing the counts in corresponding channels before integrating, the counting statistics would be improved due to the larger number of counts in each channel. In addition, it was observed that each peak is not circular as would be helpful to the integration methods, but rather is elliptically shaped due to the fact that the coincident gamma rays are of different energies and that the detectors have better energy resolution for lower energy gamma rays. This causes the peak to be narrower along one axis than along the other and gives an asymmetric peak. But, since the coincident gamma rays producing the second peak are detected in opposite detectors than are those responsible for the first peak, the two peaks have their longer asymmetric axes perpendicular to each other. Hence, if the two peaks are summed without rotation, the resulting summed peak should be more nearly symmetric than either of the single peaks. The results of utilizing this approach for the same eleven spectra from which Table 1 was compiled are shown in Table 2.

It can be seen from the data that stacking the peaks seems to improve the precision of the 3-dimensional Covell method only slightly, while significant improvements are made in the precision attainable by both the other methods, especially by the 3-dimensional Sterlinski method, where it seems reasonable to expect 1.6% precision. The statistically expected error in a peak of 4600 counts (the sum of two peaks of 2300 counts each) is approximately 1.5%. Hence, the precision of this method appears very good.

Further testing is needed, however, to determine if the method will give similar results for other data. In addition, some estimate of the accuracy of the method is needed.

FIGURE 1. BLOCK DIAGRAM OF THE ELECTRONICS
ASSOCIATED WITH MULTIPARAMETER COINCIDENCE SPECTROMETRY

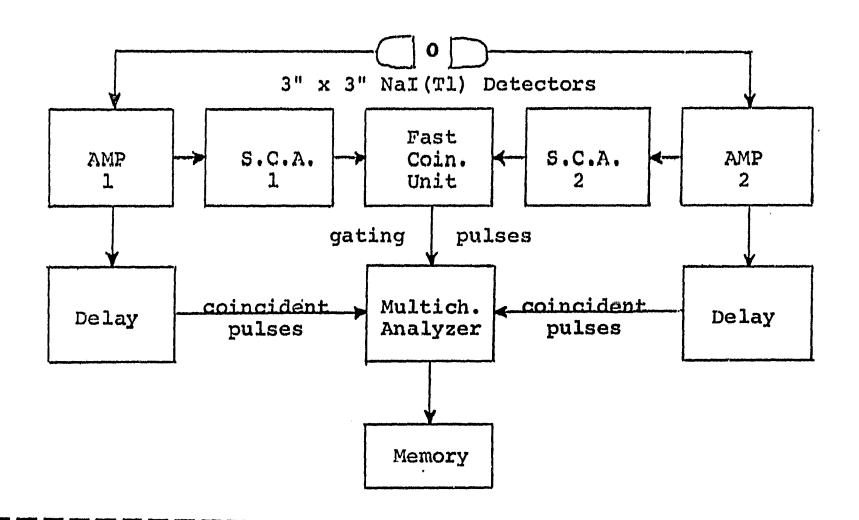
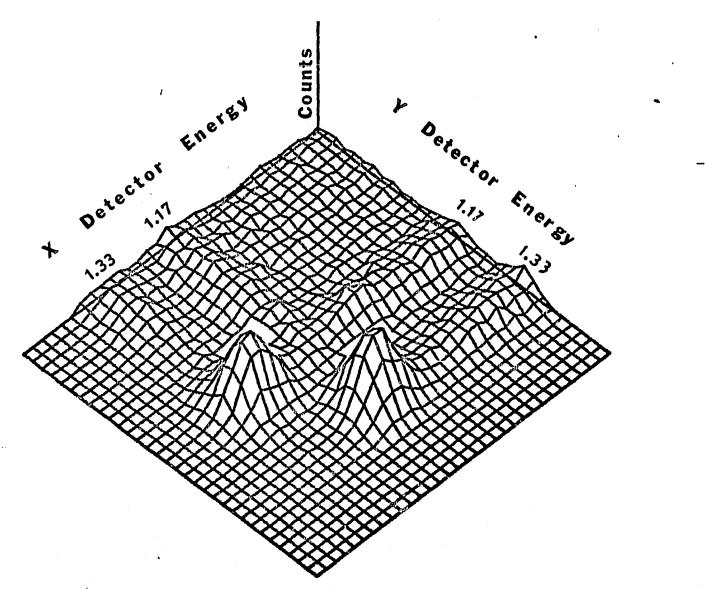
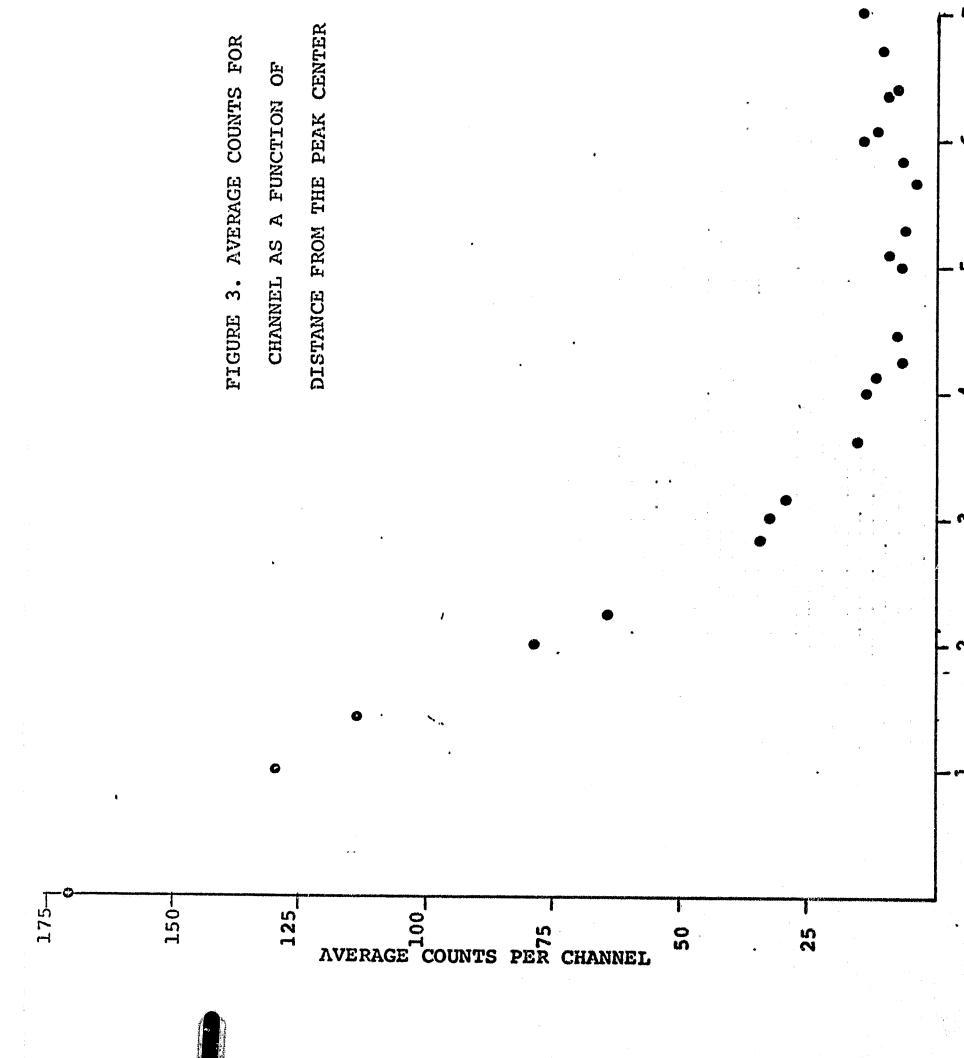
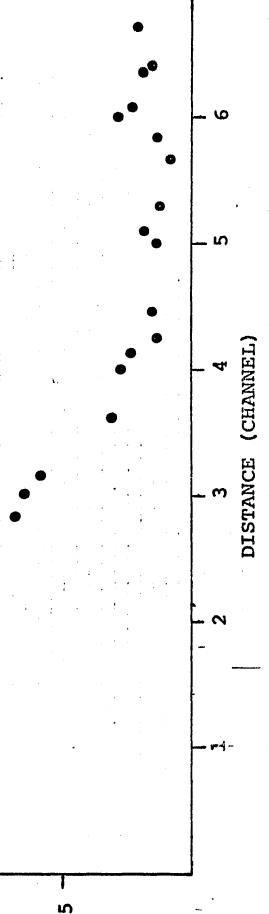


FIGURE 2. MULTIPARAMETER SPECTRUM OF 60 CO







AVERAGE COUNTS PER CHANNEL

RESULTS OF THE INTEGRATION OF A SINGLE PEAK
BY THE THREE PROPOSED METHODS

	y _n			v_n^c v_n^m			v _n		
<u>N</u>	Mean	<u>σ</u>	<u>%0</u>	<u>Mean</u>	<u> </u>	<u>%</u> 0	Mean	<u> </u>	<u>%</u> 0
3	129	38	29.2	171	54.4	31.8	150	49	33.1
5	599	72	12.0	1584	163	10.3	1178	108	9.2
7	1213	113	9.3	4805	411.	8.6	3518	276	7.8
9	1834	85	4.6	9703	440	4.5	6724	350	5.2
11	1978	101	5.1	14063	630	4.5	10521	550	5.2
13	2254	72	3.2	19713	537	2.7	15084	524	3.5
15	2211	53	2.4	24026	357	1.5	19610	540	2.7
17	2320	81	3.5	29309	823	2.8	24330	666	2.7
19	1978	151	7.7	31115	1326	4.3	28590	700	2.4
21	2402	149	6.2	39114	1634	4.2	33102	821	2.5
23	2117	197	9.3	40523	1866	4.6	36767	763	2.1

TABLE IV

RESULTS OF THE INTEGRATION OF THE STACKED PEAKS

BY THE THREE METHODS

		v _n			v_n^m			v_n^s	
<u>N</u>	Mean	<u>o</u>	<u>& 0</u>	Mean	<u>σ</u>	<u>80</u>	Mean	<u>σ</u>	<u> </u> 80
1	261	43	16.4	354	58	16.3	313	52	16.7
3	1211	99	8.2	3249	230	7.1	2421	161	6.6
5	2466	103	4.2	9805	363	3.7	7219	256	3.5
7	3708	115	3.1	19670	578	2.9	13708	380	2.8
9	4021	89	2.2	28607	613	2.1	21504	500	2.3
11	4541	132	2.9	39852	872	2.2	30676	566	1.8
13	4343	164	3.8	47877	1164	2.4	39746	650	1.6
15	4931	176	3.6	60952	1391	2.3	49427	818	1.6
17	3666	200	5.4	60241	1833	3.0	57761	899	1.6
19	4474	256	5.7	75320	2842	3.8	66215	1240	1.9
21	4094	319	7.8	79032	3084	3.9	73977	1200	1.6

References:

- 1. Covell D. F., Anal. Chem. 31, (1959) 1785-90.
- 2. Sterlinski S., Anal. Chem. 40, (1968) 1995-8.
- 3. Quittner P., Anal. Chem. 41, (1969) 1504-6.
- 4. Baedecker P., Anal. Chem. 43, (1971), 405-10.

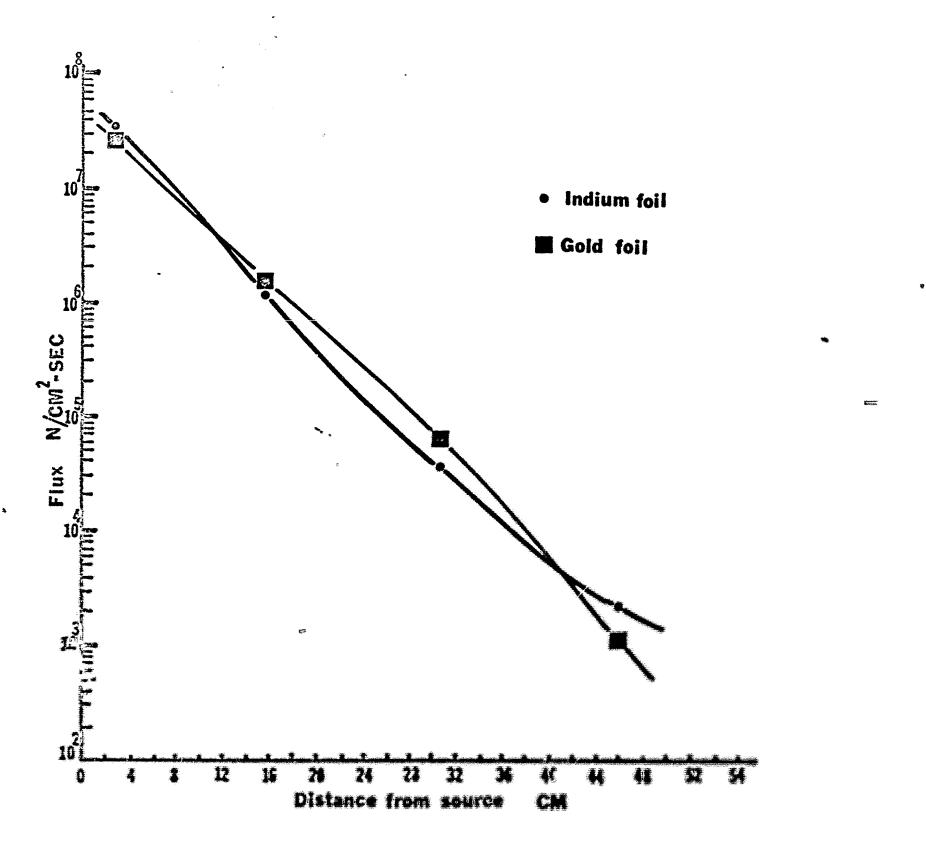
4. CAPTURE GAMMA RAY ACTIVATION ANALYSIS BY USING 252Cf

C. L. Sya

The University of Kentucky has purchased a 1.25 mg ²⁵²Cf source which was delivered at the end of April. The primary use of the source will be the development of a unique analytical technique; capture gamma-ray activation analysis for the determination of major elements in geochemical samples.

The project was divided into three stages. First, it was necessary to design and construct a facility for housing the source. The source is mounted inside an acrylic plastic tube placed in the center of two concentric polyethylene tanks (one is 250 gal. volume, the other is 275 gal.). The tanks are filled with deionized water and are surrounded with a 0.020" thick cadmium sheet. Concrete blocks provide additional shielding.

Preliminary measurements were made to determine the neutron flux using gold and indium foils with the results shown in Fig. 1.



The second step was to build the capture gamma-ray analysis system. The earliest system consisted of an 18" Lucite sidetube which could be filled with either gas or water to act as a beam port between the source and the edge of the tank. Measurements of the neutron flux showed that this arrangement was not satisfactory due to the low flux (approximately 102n cm-2 sec-1) and a 6" Lucite tube was substituted for the 18" tube. At the same time, a lead detector shield was built. The shield was designed for use with both NaI and Ge(Li) detectors. The shield consists of a cylinder of lead which provides approximately 3" of lead shielding around the detector. A square hole approximately 2" x 2" is provided for the neutron beam to pass perpendicularly through the shield in front of the detector. A sample holder is provided in front of the detector.

The whole beam path is coated with a layer of a boron carbideexpoxy mixture to absorb scattered neutrons. A 6LiF neutron screen is located in front of the detector. The inside of the detector cavity is graded with copper and tin sheet.

Trial studies were initiated by using the low neutron flux available, a 3" x 3" NaI detector and a 400 channel analyzer. Although most people in this field have used a flux of about 10 n cm-2 sec-1, some work has been done with flux of only 4 x 10 (Kusaka and Tsuji, 1970) for high cross section elements. Unfortunately, the work to date has been hampered by a high gamma-ray background due to the capture gamma-ray of hydrogen generated in the water surrounding the neutron source.

To minimize this high gamma background, a 10" diameter 9-1/2" tall stainless steel can filled with boron carbide mixed with paraffin has been constructed to act as a source holder and neutron beam collimator. The lower energy capture gamma-rays (477 KeV) from the boron should reduce the background problem.

When these construction problems are resolved, the third stage of work will consist of using the capture gamma-ray technique for the determination of a number of major elements in geochemical samples. Elements such a P, S, and Ca pose problems in conventional neutron activation analysis due to the absence of gamma-rays emitted by the radionuclides produced or the low isotopic abundance of the target nuclide. It is hoped that the capture gamma-ray analysis technique will be able to provide suitable sensitivity and selectivity for the determination of certain of these elements in rocks, soils and meteorites.

Reference:

Kusaka Y. and Tsuji H. J. Radioanal. Chem. 5, (1970) 359-367.

5. ANALYSES OF THE HOLBROOK METEORITE FOR

SILICON AND OXYGEN

W. D. Ehmann and D. E. Gillum

A series of samples of the Holbrook, Arizona chondrite which was provided by Dr. E. Gibson of the Manned Spacecraft Center, Houston, Texas, has been analyzed for silicon and oxygen by 14 MeV neutron activation. A study is being made of the changes due to weathering of these samples. One sample was recovered immediately after it fell (1912), the second, some 19 years later (1931), and the last sample was only recently recovered (1968).

Oxygen and silicon were determined by non-destructive 14 MeV neutron activation analysis according to the method described in detail by Morgan and Ehmann (1970a). These same techniques have been applied to the analysis of the returned lunar rocks (Ehmann and Morgan, 1970, Morgan and

Ehmann, 1970b).

The results are reported in Table 1 along with results obtained simultaneously on the U.S.G.S. standard rock BCR-1. The number in parenthesis is the number of analyses. The BCR-1 results are compared with those previously published for the identical BCR-1 samples by members of this group. The results for the Holbrook sample of 1912 are compared with the results of Mason and Wiik (1961). In general, the comparison of the data with previous reported data is very good.

Clearly, the Holbrook samples recovered after extensive weathering (1931 and 1968) have undergone extensive oxidation as compared to samples recovered immediately after the meteorite fall. The slight increase in the silicon abundance with weathering may be due to contamination of the specimens with ground water rich in dissolved silica, or the preferential leaching of meteoritic phases poor in silicon. The difference is small, however, and the trend may be fortuitous.

References:

Ehmann W. D. and Morgan J. W. (1970), Proceedings of the Apollo 11 Lunar Science Conference 2, 1071-1079.

Mason B. and Wiik H. B. (1961), Geochim. Cosmochim. Acta 21, 276-283.

Morgan J. W. and Ehmann W. D. (1970a), Anal. Chim. Acta 49, 287-299.

Morgan J. W. and Ehmann W. D. (1970b), Earth and Planet. Sci. Letters 9, 164-168.

TABLE V. ABUNDANCES IN THE HOLBROOK METEORITE AND BCR-1

SMPLE	THIS WORK	OTHERS
	(weight per cent)	
Oxygon		
BCR-1	46.3 ± 0.5 (6)	45.9 ± 0.5 (F)
Holbrook 1912	$35.40 \pm 0.4 (6)$	36.47 ²
Holbrock 1931	$39.7 \pm 0.8 (6)$	Note book limit
Holbrook 1968	38.6 + 0.8 (6)	pay the this
Silicon		
bur-1	25.31 ± 0.3 (3)	$25.65 \pm 0.45 (6)^{1}$
Holbrook 1912	18.56 ± 0.1 (3)	18.742
Holbrook 1931	18.91 ± 0.4 (3)	and the and
Holbrook 1968	19.40 ± 0.2 (3)	prod Mark 37-08

References for Table V.

The standard deviations listed are those calculated from replicate analyses and are for individual determinations.

¹Morgan and Ehmann (1970b).

²Mason and Wiik (1961).

6. ATTESTS OF TENTITE J2 FOR O, Si, Al, AND FO

D. E. Gillum and W. D. Ehmann

This report deals with the analysis of teltite sample J2 which was provided by Dr. D. R. Chapman of the Ames Research Center, Moffett Field, California. The specimen will also be analyzed by Dr. P. A. Schmitt, Oregon State University, and Dr. J. C. Laul, University of Chicago. Those data are provided for the use of the analysis teams.

Oxygen, silicon, aluminum, and iron were determined by non-destructive 14 MeV neutron activation analysis. The method used for oxygen and silicon has been described in detail by Morgan and Ehmann (1970a). A more general description of the techniques used including those for iron and aluminum has been given by Ehmann and Morgan (1970a). These same techniques have also been applied to the analyses of the Apollo 11 lunar samples (Ehmann and Morgan, 1970b) and Apello 12

rock 12013 (Morgan and Ehmann, 1970b).

The results are reported in Table 1 along with the results obtained simultaneously on the U.S.G.S. standard rock BCR-1. The number in parenthesis is the number of replicate analyses. The BCR-1 results are compared with results previously published by members of this group on the same BCR-1 samples. The agreement of the present standard rock data with the earlier data is very good, although the precision of the newer data is somewhat inferior. This is due to the fact that new tritium targets were used in the earlier determinations while a somewhat depleted target was used in the newer series of analyses. The lower activities obtained with the partially depleted target yielded poorer counting statistics particularly in the determination of iron and oxygen. This was especially true in the The activity produced was so low that iron determination. it was difficult to observe a photopeak from the multi-channel analyzer display of the data, but the use of computer processing of data made it possible to resolve the iron peak. levels were much superior for the silicon and aluminum determinations and the precision obtained was similar to that reported by Morgan and Ehmann (1970b) for analyses of BCR-1 and lunar rocks.

References:

Ehmann W. D. and Morgan J. W. (1970a), Proceedings of the Second Oak Ridge Conference on the Use of Small Accelerators for Teaching and Research, U.S.A.E.C. Report, CONF-70032, 205 220.

Ehmann W. D. and Morgan J. W. (1970b), Proceedings of the Apollo 11 Lunar Science Conference 2, 1071-1079.

Morgan J. W. and Ehmann W. D. (1970a), Anal. Chim. Acta 49, 287-299

Morgan J. W. and Ehmann W. D. (1970b), Earth and Planet Sci. Letters 9, 164-168.

TABLE VI. ABUNDANCES IN TEKTITE J2 AND BCR-1

SAMPLE	THIS WORK	MORGAN AND EHMANN (1970b)
Oxygen		
BCR-1 (1824)	$46.0 \pm 1.0 (6)$	45.8 ± 0.8 (6)
BCR-1 (1825)	$45.7 \pm 0.9 (7)$	$45.7 \pm 0.4 (6)$
BCR-1 (1826)	$46.7 \pm 1.1 (7)$	45.9 ± 0.5 (6)
BCR Mean	46.1 ± 0.67	45.6 ± 0.42
Tektite J2	49.1 ± 1.9 (8)	internal Albana -
Silicon		
BCR-1 (1825)	25.69 ± 0.41 (8)	25.51 + 0.22 (6)
BCR-1 (1826)	$26.14 \pm 0.32 (8)$	25.75 ± 0.45 (6)
BCR Hean	25.92 ± 0.21	25.63 ± 0.20
Toktite	$32.73 \pm 0.62 (8)$	stops design
Aluminum		
BCP-1 (1825)	7.04 ± 0.27 (3)	7.0 \pm 0.1 (3)
Tektito	7.52 ± 0.17 (3)	₩6. +46.
Izon		
KI-1	$9.7 \pm 0.5 (3)$	$9.6 \pm 0.2 (3)$
Toktite	$6.4 \pm 0.6 (3)$	### AND

The standard deviations listed are those calculated from replicate analyses and are for individual determinations.

B. List of Capital Equipment Purchased

Capital equipment purchased during the period January

1, 1971 - December 31, 1971 is listed below:

U.S.A.E.C. Funds-

1. Surface Barrier Detector, ORTEC \$345.00

2. NIM Bin and Power Supply,

Harshaw Model NH-84A

575.00

3. FET Preamplifier, ORTEC Model 609A

385.00

Total = \$1305.00

(*Instruments or equipment having a useful life of over one year and a cost exceeding \$100.00.)

University of Kentucky Funds-

1. Encapsulated ²⁵²Cf neutron source (1.25 mg.)

\$15,960.00

Note: The University of Kentucky was the first purchaser of 252 Cf. This source is used in research described in Section V-A-3 of this report.

C. Academic Progress

Research supported by this contract was included in the Ph.D. dissertations of Dr. David E. Gillum and Dr. Patricia M. Santoliquido. Each received their degree during the 1971 Contract year. One M.S. candidate and two Ph.D. candidates are continuing research initiated under support from this contract.

D. Incident Report

No incidents, as described in "attachment A" have occurred on this project, during the period of this report.

E. Federal Agency Report Statement

The Principal Investigator also held N.A.S.A. Grant NGR 18-001-058 during the period covered by this report. The N.A.S.A. grant was operated with the services of a full time Postdoctoral Associate and dealt specifically with analyses of lunar samples and not technique development. This U.S.A.E.C. Contract specifically supported technique development in the field of activation analysis and application in the study of the geochemistry of meteorites. The roles of the two contracts were independent, however, many techniques developed under support of this U.S.A.E.C. Contract were later applied to specific problems encountered with the lunar samples. Publications supported exclusively by the N.A.S.A. Grant are not listed in Section II, but a list of reprints will be provided on request. The Principal Investigator received 50% of his summer salary (2 months) from the U.S.A.E.C. Contract and 50% from the N.A.S.A. Grant.

VI. ACKNOWLEDGEMENT

The support of the U.S.A.E.C. over the eleven years of this project's work is gratefully acknowledged. The excellent support of the University of Kentucky in providing funds for the 14 MeV neutron generator facility, the ²⁵²Cf source, and the many items of equipment required to supplement those provided by this Contract is also gratefully acknowledged. The author also wishes to express his gratitude to the staff of the University of Kentucky Research Foundation for their assistance in contract management and accounting.